

**NASA-CR-205684**

*Final  
Report  
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10/1/97*

## **PROJECT CLOSING REPORT**

### **“DURABILITY AND LIFE OF CERAMIC MATRIX COMPOSITES IN COMBUSTION ENVIRONMENT”**

**(NCC-3-205)  
(Project Period: 6/15/91-11/30/96)**

Submitted to:

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**Sept 24, 1997**

## **DURABILITY AND LIFE OF CERAMIC MATRIX COMPOSITES IN COMBUSTION**

### **ENVIRONMENT**

#### **INTRODUCTION**

It is now generally recognized that the next frontier in the commercial air travel will be achieved by an optimum combination of the concepts being used for the air and space transports. It will depend upon the new engines specifically designed to meet the demanding strength, temperature, environment and economy requirements. The successful development of an advanced propulsion system for a 21st Century High Speed Civil Transport will require key advances in the combustor system. Minimizing nitrous oxide ( $\text{NO}_x$ ) emission is critical, since the deleterious interaction within the ozone layer would occur at the proposed vehicle cruise level of about 75,000 feet. In order to achieve the low  $\text{NO}_x$  levels an efficient combustor operating at controlled equivalence ratios and high combustion temperatures are required. This requires a combustor, whose one portion will be operating in an oxidizing environment while the other portion will operate in a reducing environment. This rather unusual requirement has introduced considerable challenge to the materials scientists and engineers, the development of material systems capable of long-life at high temperatures (up to 3000F) in an oxidizing and/or reducing environment.

This research was aimed at evaluating the environmental durability of potential material systems which have been identified for use in the fabrication of a combustion liner for the High Speed Civil Transport propulsion system. The leading material systems for this application were silicon-based ceramics (silicon carbide, silicon nitride and molybdenum di-silicide and their composites). In addition, alumina-based ceramics and rare earth stabilized zirconia systems was also planned. Degradation of these material systems in both a fuel-lean (oxidizing) and a fuel-rich

(reducing) environment has been an important issue, particularly with the anticipated 18,000 hour lifetime.

It was anticipated that these materials will have various degradation modes at such high temperatures and long use times. Under this research the study on the interaction of oxygen, hydrogen and water vapor with composites and composite constituents (fiber and matrices) at high temperatures under a range of operating pressure conditions was carried out.

### MAIN ACCOMPLISHMENTS

In order to understand the formation of protective scale morphology fundamental furnace studies of the scale growth, composition and morphology on various silicon carbide and silicon nitride matrix composites, as well as pure monolithic materials, were performed in inert, oxidizing and reducing environments for varying times and temperatures. Scale composition and physical nature (e.g. scale thickness, uniformity) were used to aid the elucidation of the mechanics of scale formation.

Kinetic studies were conducted by recording the weight change with time for these materials in the various environments in order to establish the rates of scale formation and substrate recession. Microstructural and phase changes were identified in order to confirm or refute the thermodynamically predicted reduction mechanisms.

Burner rig studies were carried out to test the materials under the high temperature, high velocity, thermal cycling conditions. Testing were performed in a fuel-lean environment of the High Pressure Burner Rig. Post-test microstructural characterization of the materials with burner rig operational/test parameters were performed in concert with the Lewis Research Center facility materials engineers.

In addition the residual mechanical behavior was examined to understand the effects of environmental degradation on the strength of the ceramic matrix composites.

The effect of water vapor on the oxidation of SiC was of great interest since water vapor is a combustion product and is thus found in all combustion environments. It was demonstrated that water vapor causes rapid degradation rates of SiC at concentrations close to those expected in combustion environments. Using high temperature furnace thermogravimetric oxidation studies of SiC with a flowing atmosphere of oxygen and water vapor under controlled conditions, the degradation was attributed to two mechanisms which operate simultaneously. First, it was shown that water vapor partial pressures of about 0.1 atm and higher enhance the parabolic oxidation rate of SiC compared to the rates observed in 1 atm of oxygen. It was demonstrated that the oxidation rate was linearly dependent on the water content above the threshold value of about 0.1 atm water vapor. This enhanced oxidation rate was attributed to the higher solubility of water vapor in the silica scale compared to oxygen. Apparently the diffusivity of water vapor in the silica scale controlled the oxidation rate of SiC.

The second degradation mechanism of SiC in water vapor was attributed to the volatility of the silica scale formed during oxidation. The rates of silica volatilization were measured as a function of temperature. Very little temperature dependence for this process was observed. A parabolic rate law was found to describe the SiC oxidation and simultaneous volatilization of silica. Experimentally determined weight changes were fit to an expression for parabolic kinetics to determine the rate constants for each mechanism. At long times (several hundred hours), a steady state process was predicted to occur. One would therefore expect that SiC would oxidize at the same rate as silica volatilizes resulting in an oxide of constant thickness on SiC which is linearly recessing. Thus, the consumption of SiC was controlled by the volatilization rate of silica.

The implications of this parilinear degradation mechanism for the use of SiC in combustion environments were investigated. An expression based on temperature, total pressure, partial pressure of water vapor, and gas velocity was developed to predict SiC recession rates under actual combustion conditions which occur at higher pressures. By this expression, recession rates were found to be rapid, so that in a combustion environment insufficient lifetimes for thin SiC components are predicted. These predictions were confirmed by high pressure burner rig testing conducted at NASA-Lewis.

In other work, the oxidation rates of SiC and  $\text{Si}_3\text{N}_4$  were measured side by side in a clean environment. The oxidation rate of  $\text{Si}_3\text{N}_4$  has generally found to be slower than that of SiC. This difference has been attributed to the formation of an inner oxide layer of  $\text{SiO}_x\text{N}_y$ . It was found in this study that the oxidation environment can significantly affect the oxidation rates. The importance of this issue has not been previously carefully addressed in the literature. The results from the clean environment were evaluated to clarify some of the outstanding questions in the literature regarding the oxidation mechanisms of these materials.